Notebook Series -- Notebooks by Experimenters Other Than Edison
Phonograph Record Experiments -- Record Varnish Books
Notebook, N-14-05-14

This notebook was used during May 1914 by an assistant in the chemical laboratory named Davis (possibly W. B. Davis, author of N-20-07-27 [unselected]). The entries relate to experiments to prevent wave formation on records while varnishing. Most of this work involved the use of various resinous compounds, particularly phenolic resin. Some entries refer to a "Mr. T. A. E. idea," while others indicate that something was given to Edison for further experimentation. The flyleaf is inscribed "Note book of Mr. Davis." The front cover is marked "Davis." The book contains 5 numbered pages followed by approximately 25 unnumbered pages.

150° F seems to be the maximum.

Now with standard OOC (or one of similar strength) and applying air. ~~the pull is maximum before limit~~.

Slip mean. of a few drops to the surface, then pull a 4" vacuum. for ½ hour. then rise to 100° F for ½ hour. then take directly to 200° F for 1 hour.

~~In an hour~~

In practice the ~~waves~~ waves begin to form at about 140° F — 1½ hour

May 5 - 14

Mr. Y.A.S. idea

Flow and stand for about 1 hour.
Then cover rack with a wire gauze
and heat, at the same time drawing ½" vacuum.

1.00 P.M. - 120°F.
2.05 " - 120 "
2.30 " - 124 "
3.00 - 124 "
3.30 - 124 "

May 5 - 14

Trying to dry the phenolic resin, which is in alcoholic sol. and flowed over nickle plate.
at present these films are flowed ~~and~~ allowed to stand for about 1 hour, then put into a dryer and heated for 7 hours at about 230° F. On the removal of these plates from the dryer the resin film has dried in a wave.
The object of these experiments is to prevent the wave formation.

The average viscosity of the resin solution = 4.00
The diameter of the disk =
The volume of alcohol resin solution on 1 disk =
The thickness of the dry resin film =

(1) The object of this experiment is to remove the alcohol, as quickly and as thoroughly as possible before applying heat.

Flow and transfer directly to vacuum. pull ½" for ½ hour
pull ½" for ½ hour,
" 2" for ¼ hour
" 5" for ½ hour.
" 10" for ¼ hour
" 30" for ½ hour
" 26" for ¼ hour.
Then heat for 1 hour 100°F. - 130°F after which gradually increase heat and observe at different stages say every 15 minutes -

May V-13

(2) Two plates which had been flowed, and stood for two days.
On to 1 was put a thin layer of Machine oil.
put into oven at a temp of 140°F at 3.50
230°F at 11.00

Heated for 15 minutes between 230-240°F, this length of time rendered them perfectly hard, when cool.
If any difference, I should say, the one without oil were the better of the two,

(3) With the object of lowering the B.P. of the alcohol, took 20cc of the resin solution, and diluted with 10cc CS_2 (allowed to stand over-night

Heated to 50°C - 8.50
70°C - 9.10
170°F - 9.35 OK
200°F - 9.45 Traces of riffles showing
245°F, 10.05 Mar traces

But developed riffles on rendering removing from heat.
They should be allowed to cool in apparatus gradually.

(3a) The same as (3) only allowed to cool in bath gradually.
Started - 11.00 a.m. - allowed to stand until 2.00 - 3 hours
Then heated as above, and allowed to cool without removing from the oven. Started 1.00 to heat finished at 2.20 at a temp of 240°F

(4)

May 6 - 1[illegible]

Flowed 6 plates with resin which had stood since [illegible] night.
" 4 " " fresh solution
allowed to stand for 1 hour.
Transfered to vacuum -

9[illegible]	- pulled 1/4"	- 1/2 hour	3 hours
9[illegible]	- " 1"	- 1/2 hour	
10[illegible]	- " 10"	- 1/2 hour	
10[illegible]	- " 20"	- 1/2 hour	

Turned off vacuum pump.
and heated for 1 hour at 120-130 - Started 11[illegible] — 4 hours.
" " " 12[illegible]
170-180 - " 12[illegible] — 4 1/2 hours
220 — " 12[illegible] — 5 hours.
1[illegible] Turned off heat
Total time 5 hours

Turned off heat and allowed to remain in oven to anneal until fairly cool.

The plates which were flowed with the solution which had stood over night were poor.
Those which were flowed with new solution were fairly good

This experiment was performed with the idea that the disks should be annealed.

after standing over night these good ones proved to wrinkle very badly.

(5)

Flowed with regular varnish and allowed to stand for 24 hours. put into an oven at 230° F. and allowed it to remain for ½ hour. cooled in oven. (ng)

(5a) heated some with water in a watch glass. and [redacted] heated,

(6) Flowed a plate with 20cc of varnish allowed to drip and heated directly to the desired temp. of 230° F. (ng)

(7) Flowed with 2% Copal added, allowed to stand 24 hours then heated.

(8) Flowed with 2cc Pine added, allowed to stand 24 hours then heated.

The preparation of p-chloro-aniline - $C_6H_4Cl\,NH_2$ - (1:4)

1- mono-chloro-benzene - C_6H_5Cl -
By the action of Cl on benzene in presence of a carrier.

Take the 2 the 1 and material and can be charged for 15 hr.

2- p-chloro-nitro-benzene - $C_6H_4Cl(NO_2)$ (1:4)

This together with the o- compound is formed by the nitration of mono-chloro-benzene.

3- p-chloro-aniline - $C_6H_4Cl(NH_2)$ 1:4

This aniline is formed by the reduction of p-chloro-nitro-benzene.

$C_6H_4Cl\,NO_2 = C_6H_4Cl\,NH_2$
157.5 = 127.5

Therefore 100 gm of p-chloro-nitro-benzene should give theoretically 80 gm of p-chloro-aniline.

May 9-10

Preparation of p-chloro-aniline

Nitrated mono-chloro-benzene
150 gm Con. H_2SO_4
100 " " HNO_3
50 " C_6H_5Cl.

Nitrated for 1 hour at 60°C.
Nitro compound [illegible] amount of the [illegible] in product.

From the follow formula:-

$$C_6H_5Cl + HNO_3 = C_6H_4Cl\,NO_2$$
$$112.5 + 63 = 157.5$$

Therefore 100 gm of mono-chloro-benzene should theoretically give 140 gm of the mixed nitro compounds.

This experiment was performed with just Con. HNO_3

From the above equation 50 gm C_6H_5Cl would require exactly 90 gm of Com. HNO_3 of 1.42 sp. gr.

Took 50 gm C_6H_5Cl } heated for 1 hour, in boiling
100 " Con. HCl } water, hardly any nitro-compound was formed.

(Mg.)

Took – 50 gm C_6H_5Cl
100 " fuming HNO_3
nitrated in the cold, –
yield = (about) 50% of theory of mixed compounds.
Total weight of nitro-compounds = 37 gm

(A)

[illegible] gm. fuming HNO_3
300 gm. Strong [illegible]
[illegible] cool
[illegible]
[illegible] —
[illegible] cool, and filtered
[illegible] in the [illegible]
[illegible] weighed = 147 gm this co
[illegible] pure p-Dichloro-nitro-benzene the
melting point of the pure compound = 83.5°C
[illegible] mixed compound =

Took 50 gm of the (B) [illegible]

50 gm compound
50 " Tin
200 " [illegible]
added [illegible]
Then added [illegible] of 50 gm [illegible]
[illegible]
and distilled with steam.
[illegible] 37 gm —

Took 50 gm of (B) [illegible]

[illegible] fractionally

1st fraction M.P. –
2nd " " –
3rd " " –

Neutralizing the free acid in the form of Phenol-Sulphonic Acid

May – 18 –

	75.9285
	73.8135
	2.1250 $BaCO_3$
Weight of $100\ SO_3$ –	16.5000
	38.0059
	8.4941

To 100 gm add 2.3 gm $BaCO_3$

May – 19 –

	73.1235
	72.8300
	.2935
Weight of	30.9257
	24.8300
	6.0957

To 100 gm add 1.0 gm $BaCO_3$

After standing 3 days – Started May 15 – 18

Solubility of o-phenol. Sulphonic acid in 95% alcohol

	21.0230
	17.9052
	3.1178

Preparation of [illegible], naphthalene sulphonic acid.

18.2755
17.9052
.3703

20% — 95% strong

1.6% Dilute

Preparation of α-phenol-sulphonic acid

equal parts phenol and [illegible], mixed in an [illegible] which is placed [illegible] and [illegible] in a [illegible] a four [illegible]

Barium of $BaCO_3$ [illegible] the [illegible] acid.

May 21 $BaCO_3$ 78.3500
75.7540
2.6060

[illegible] 94.2700
18.7083
5.5617

Eqv. 45.4 per 100

May 26 $BaCO_3$ 75.7540
72.8032
2.9508

[illegible] 95.7930
18.7083
7.0847

Eqv. 47.5 per 100

375 gm. acid eqv. 155 gm $BaCO_3$
175 gm. BaO

Indigo-mono, and di-sulphonic acid -

Solubility of Indigo-monosulphonic acid in 95% alcohol

$$\frac{15.1185 - 15.0240}{.0945} \text{ in } 20^{\circ}C = .47\% \text{ sol in } 95\% \text{ alcohol}$$

Solubility of Indigo-disulphonic acid in 95% alcohol.

$$\frac{15.2427 - 15.0240}{.2187 \text{ in } 20^{\circ}C} = 1.09\%$$

Benzene-Disulphonic acid -

2 parts Benzol } Warmed gently, then allowed to stand
2 " Con. H_2SO_4 } over night.

Diluted the unabsorbed benzol, and neutralized the free acid in cold with $CaCO_3$, and formed the Pb salt of the very insoluble

Decomposed this by means of the H_2S in the water-bath, and obtained a large yield of the acid.

The free acid dissolved very slowly in the water-bath, with either water or alcohol on the outside, and there was a slight excess of H_2S present.

(This might not do so if the H_2SO_4 was neutralized ?)

Solubility of Benzene-sulphonic acid in 95% alcohol.

$$\frac{23.2850 - 15.2500}{8.1185} \pm 20^{\circ}C$$

45.5% Sol

$C_6H_5SO_3H + H_2SO_4$
364 + 98

To 15 gm of the Pb salt added 4.5 gm of 95% H_2SO_4 gradually with stirring & 50% more % H_2SO_4 was used. The was put over the Pb salt, and with two drops filtered.

Remaining Sulphuric - From 15 gm Material

22.8600

$C_6H_5SO_3Pb$ — $C_6H_5SO_3H$
~~[illegible]~~ — 158

Therefore to make a 10% alcoholic solution from the Pb. salt, take 32 gm Pb. Salt & 102.00 gm 95% alcohol.

(To the mass of 95% alcohol is added 6.2 gm H_2SO_4. This is the required amount to ~~form the~~ decompose the Pb salt and free the acid.)

Not 5 gm. H_2SO_4 only as above. 95% H_2SO_4 — Thus 5.15 gm of the Com. Conc. H_2SO_4 is required.

By having a large quantity:

[illegible] gm Com. H_2SO_4 heated gently for 12 hours [illegible] Benzol [illegible] Sulfonic Acid [illegible] the undissolved benzene, which weighed

(Crude Comp. 720 gm Took — 600 gm H_2SO_4
Remaining Benzol 393 " " — 400 " Benzol)

Therefore 107 gm of Benzol was used.

$C_6H_6 + H_2SO_4 = C_6H_5SO_3H$
78 ——— 158

Therefore from the 107 gm of benzol used I should get about 216 gm of $C_6H_5SO_3H$
or 497 " of $(C_6H_5SO_3)_2Pb$.

$PbCO_3$ — .05 ½ lb. — get back $PbCO_3$ — .05 lb.
H_2SO_4 — .01 lb. —
~~C_6H_6 — .03 lb.~~ — C_6H_6 — .03 gal
C_2H_5OH — .04½ lb.

$H_2SO_4 + PbCO_3$
98 367 — 600 lb. resp. 2247 lb. $PbCO_3$

600 lb. — H_2SO_4 — .01	—	$ 6.00
107 lb. — C_6H_6 — .03	—	3.21
2247 lb. — $PbCO_3$ — .05	—	112.35
		121.17

Not figuring the small amount of H_2SO_4 required to liberate the free acid from the Pb. salt.

1 lb. Cost .58¢

[illegible] acid – think 20 cc

15.9835	15.7575	15.4585
13.4127	13.4127	13.4127
2.5708	2.3448	2.0458

$(C_6H_5SO_3)_2Pb + 3H_2O) + H_2SO_4 = 2C_6H_5SO_3H + PbSO_4$

575 + 98 = 316 + 303

The amount of Pb. salt to take to make a 10% solution of the acid 316 : 575 : 10 : 18.25 Pb. salt to 100

(I think 18.5 gm per 100 cc)

The amount of acid to dissolve in 100 alcohol to liberate the acid from 18.25 of the Pb salt.

575 : 95 : 18.25 : 3.25 of Con. H_2SO_4

(I think 3.5 gm per 100 cc)

Made 500 cc

92.5 gm $(C_6H_5SO_3)_2Pb$
17.5 gm Con. H_2SO_4
500 cc Alcohol (denatured)

approximate 10% solution – given to Mr. J.A.E.

19.6730
15.0240
4.6490

Equal weights of Con. H_2SO_4 and [illegible] to stand – 300 gm of each [illegible]

Weight of acid [illegible]	=	360 gm
Weight of [illegible]	=	240 gm
		600 gm

Therefore 100 gm Con H_2SO_4 Oxyfluorated 20 gm C_6H_6 in 8 hours heating and 48 hours standing

$C_6H_6 + H_2SO_4$ = [illegible]
78 + 98 = 158

78 : 158 : 20 : 40.5 gm $C_6H_5SO_3H$ to 100 acid

78 : 98 : 40.5 : 50.8 gm H_2SO_4 to form $C_6H_5SO_3H$.
49.2 gm. H_2SO_4 unaffected.

$H_2SO_4 + PbCO_3 = PbSO_4$
98 + 367

98 : 367 : 49.2 : 184.3 gm $PbCO_3$ to neutralize free acid

$2C_6H_5SO_3H + PbCO_3 = (C_6H_5SO_3)_2Pb.$
316 + 367 = 521

316 : 367 : 40.5 : 47. gm to form the Pb. salt.

$(C_6H_5SO_3)_2Pb\ 3H_2O + H_2SO_4 = 2C_6H_5SO_3H$
575 + 98 = 316

575 : 98 : 73.6 : 12.5 gm acid to free Oxyfluoric Acid

Amount of Con. H_2SO_4 taken ——— 100 — @ — .01 d.h.

Amount of C_6H_6 ~~taken~~ used ——— 20 — @ — .03 d.h.

Amount of $C_6H_5SO_3H$ formed ——— 40.5

Amount of $PbCO_3$ used for neutralizing free acid — 204.3 — @

Amount of $PbCO_3$ used to form $(C_6H_5SO_3)_2Pb$ — 47.0 — @

Amount of H_2SO_4 used to combine with the Pb of the $(C_6H_5SO_3)_2Pb$ liberating the free acid — 12.5 — @ — .01 d.h.

Amount of Pb. Sect formed ——— 73.6

~~$2C_6H_5SO_3H + PbCO_3 = (C_6H_5SO_3)_2Pb + 3H_2O$~~

$2C_6H_5SO_3H + PbCO_3 = (C_6H_5SO_3)_2Pb + 3H_2O$

316 + 367 = 575

367 : 575 : 47 : 73.6 gm of the Pb salt contain H_2O

I have used a total of 231.3 gm $PbCO_3$

$PbCO_3 + H_2SO_4 = PbSO_4$

367 + 98 = 303

367 : 303 : 231.3 : 190.9 lbs. $PbSO_4$ left

A solution in alcohol is not what's wanted.

Amount of H_2SO_4 used ——— 100
Amount of C_6H_6 used ——— 20
Amount of $PbCO_3$ used to neutralize free acid — 204.3
Amount of $C_6H_5SO_3H$ formed ——— 40.5

OK

Heated the mixture on the H_2O-bath for 9½ hours, then allowed to stand over night, separated in the morning, diluted the acid syr. with about 10 times its volume of H_2O, and neutralized the free acid with the calculated amount of $PbCO_3$ required, filtered and concentrated the filtrate on the H_2O-bath until a large crop of crystals were formed these on standing rapidly resumed the liquid state, due to their exceedingly hydroscopic property.

over

100 H_2SO_4 dissolved 20 C_6H_6

$H_2SO_4 + C_6H_6 = C_6H_5\text{-}SO_3H$
$98 + 78 = 158$
$78 : 98 : 20 : 25.1$

} 20 C_6H_6 combined with 25.1 H_2SO_4

There was 100.0 H_2SO_4 taken
25.1 " combined
74.9 " free to be neutralized with $PbCO_3$

$H_2SO_4 + PbCO_3$
$98 + 267$
$98 : 267 : 75 : 204.3$

} The 75 H_2SO_4 requires 204.3 $PbCO_3$ for neutralization

1 naphthol-2-sulphonic acid – $C_{10}H_6OH\,SO_3H$

a-naphthol – 1 part
Con. H_2SO_4 – 2 parts

} heated on water-bath for 4 hours

dilute with H_2O, neutralize the free acid with the required amount of $PbCO_3$, and filter. Concentrate the filtrate on the water-bath, and then pour into a saturated sol. of KCl. (which is hot.) when the acid separates out.

Solubility in 95% alcohol.

2 - Naphtol - 3 - Sulphonic acid

B - 2 Naphtol - 6 Sulphonic acid - Beilstein - 2 - 869
2 Naphtol - 3 Sulphonic Acid - Watts

B - Naphtol — 1 part
Con. H_2SO_4 — 2 parts
} heated on water-bath for 1 hour

$C_{10}H_7OH + H_2SO_4 = C_{10}H_6OHSO_3H$
144 + 98 = 224

Took { 30 gm $C_{10}H_7OH$
60 gm H_2SO_4 } 144 : 98 : 30 : 20.4 gm H_2SO_4 reqd.

60 gm H_2SO_4 taken
20 " " used
40 " " to neutralize.

$H_2SO_4 + PbCO_3$
98 + 267
98 : 267 : 40 : 109 gm $PbCO_3$ to neutralize the free acid

Alizarin Sulphonic acid - $C_6H_4(CO)_2C_6H(OH)_2SO_3H$

Heat = parts alizarin with fuming H_2SO_4 at 130 - 140
Orange yellow crystals soluble in alc. and H_2O

If no separation occurs from the acid sol.
neutralize the free acid with $PbCO_3$ - filter
Then add $PbCO_3$ to the filtrate forming the Pb salt which is insoluble in H_2O
This Pb salt with the excess of $PbCO_3$ is decomposed with H_2SO_4 (which is in a small amount of H_2O) and the acid try to crystallize from this aqs. sol.